

APPENDIX A

1. PURPOSE

The following measurements were performed to specify a structure of a denatured polyvinyl alcohol described in the Examples of the present invention.

2. ANALYTICAL METHOD

2-1. SAMPLES AND PROCESS

Prepared samples were dissolved in dense dimethylsulfoxide (70°C) for a ^{13}C -NMR(BCM) measurement and also for a ^{13}C -NMR(DEPT) measurement. Methanol was added by about 0.5% to a part of the samples before the ^{13}C -NMR(BCM) measurement, since the samples might contain methanol as one of the original components.

2-2. ANALYZER

FT-NMR apparatus: LA400 (JEOL)

2-3. MEASUREMENT CONDITIONS

Concentration: about 10%

BCM: proton complete decoupling measurement

DEPT: 135° (methine and methyl face upward while methylene faces downward; quaternary carbon disappears)

Conditions for the measuring apparatus are described in the charts.

3. RESULTS AND CONSIDERATION

- 1) Chart No. 1 shows results of ^{13}C -NMR measurement on the samples.

In the measurement, PVA as a main component was recognized together with polyvinyl acetate (VAC) and 2-substitutional benzene. The VAC was regarded as an unsaponified part of the PVA, and the 2-substitutional benzene was considered as a structure having an amino group at the 0-position on the basis of the chemical shift value. Uncertain peaks are formed at 73.5 ppm, 68.3 ppm, 48.3 ppm and 38.3 ppm, and these peaks are considered as being provided by the structure combining the 2-substitutional benzene and a main chain, though it was substantially impossible to analyze the structure.

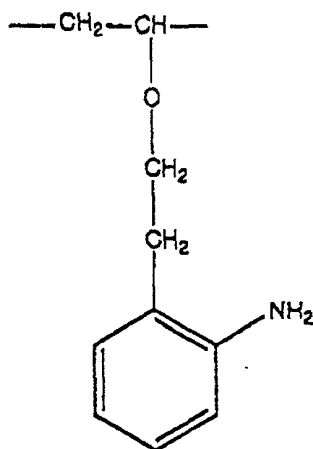
- 2) Chart No. 2 shows results of ^{13}C -NMR (DEPT) measurement on the samples.

The results show that the respective 73.5 ppm and 38.3 ppm in the uncertain peaks of Chart No. 1 denote methylene, 68.3 ppm denotes methine and 48.3 ppm denotes either methine or methyl.

3) Chart No. 3 shows results of ^{13}C -NMR measurement on the methanol-containing samples.

In the measurement, the 48.3 ppm peaks recognized in the above 1) and 2) were consistent with the methanol peak. It was determined accordingly that the 48.3 ppm peak denotes methanol that was included in the original samples in an order of several thousands of $\mu\text{g/g}$.

4) The structure of the part that the main chain and 2-substitutional benzene were combined was estimated as shown below on the basis of the above 1)-3). However, it still remains in the realm of speculation as the chemical shift value cannot be determined.



[Charts]

Chart No. 1-1: ^{13}C -NMR (BCM) measurement result for samples

Chart No. 1-2: Enlarged spectrum of Chart No. 1-1

Chart No. 1-3: Enlarged spectrum of Chart No. 1-1

Chart No. 2-1: ^{13}C -NMR (DEPT) measurement result for samples

Chart No. 2-2: Enlarged spectrum of Chart No. 2-1

Chart No. 3-1: ^{13}C -NMR (BCM) measurement result for samples including methanol

Chart No. 3-2: Enlarged spectrum of Chart No. 3-1

Experimental data US patent application Serial No. 09/452,731

1-1-1-1

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Sample: 111111
 Solvent: 111111
 Purity: 111111
 Name: 111111

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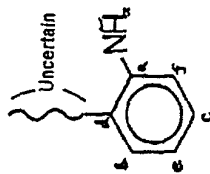
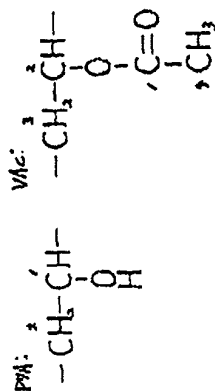
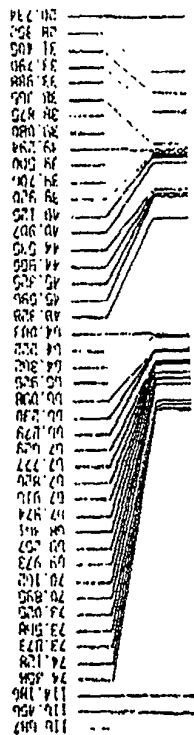
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Chart No. 1-1



e: peak caused by PVA

methanol

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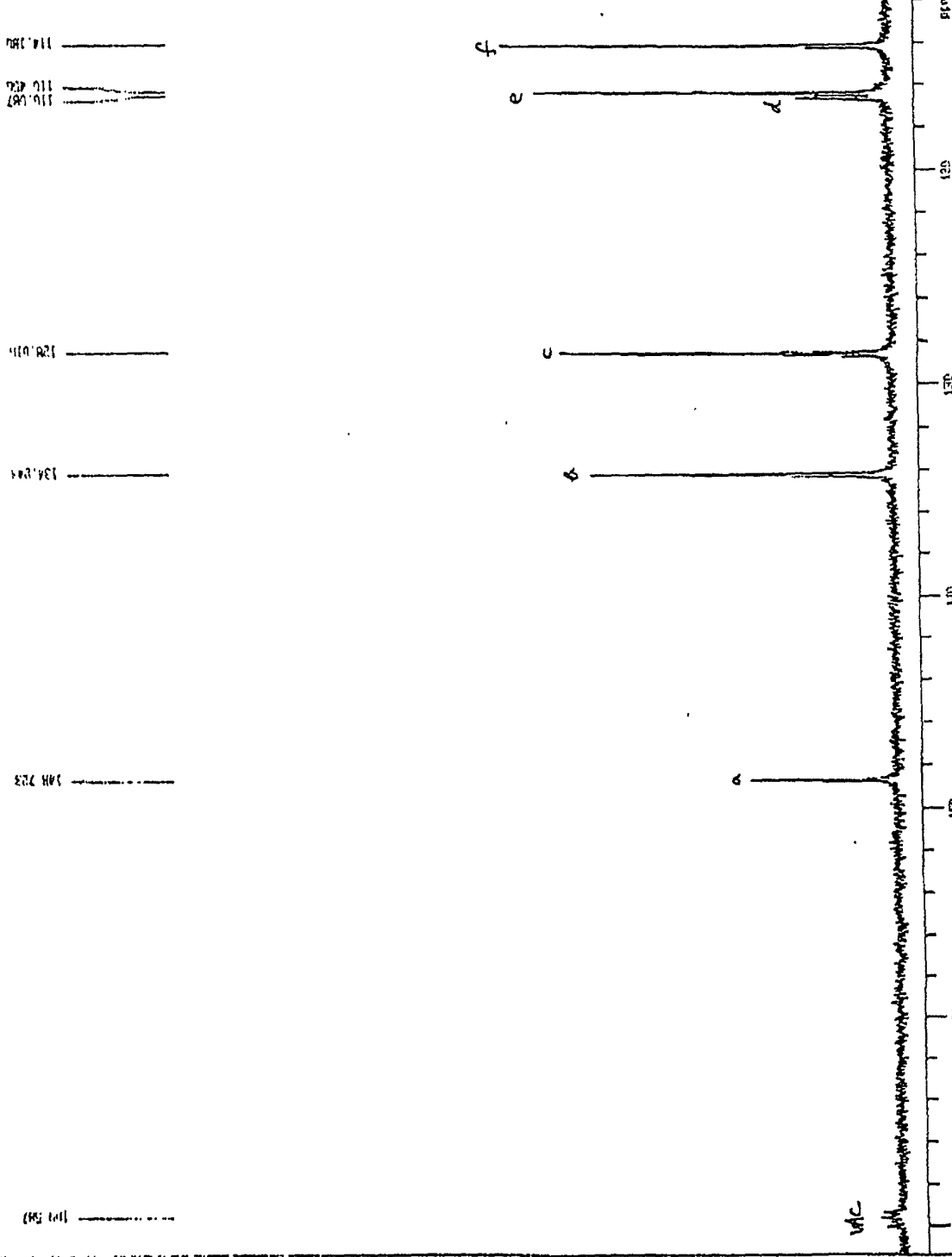
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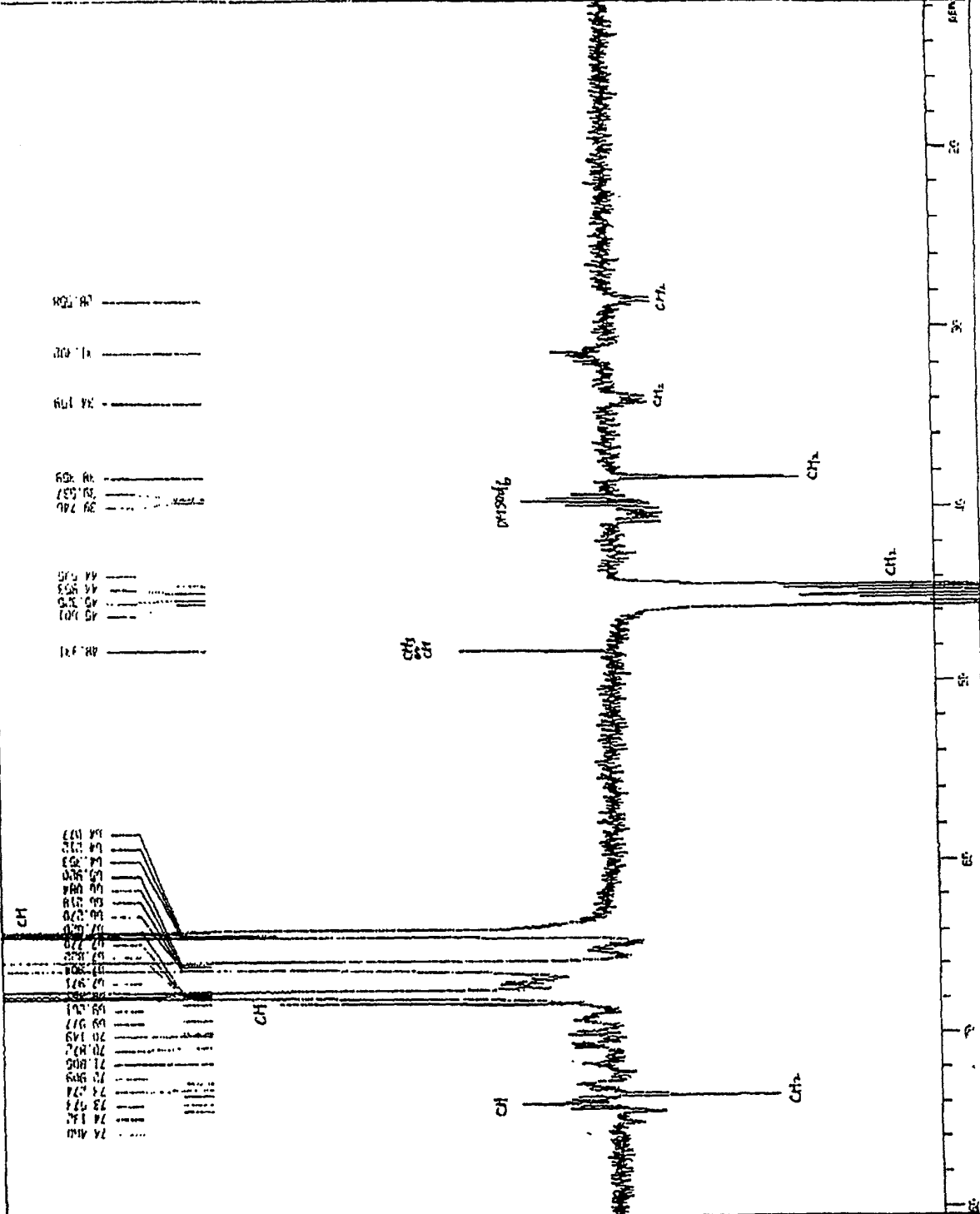
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 Cyclic: 1 Line
 Pulse: 1.00 Hz
 Residual: 21
 Observed: 132
 Observed: 132.40 MHz
 Observed: 132.40 MHz
 Solvent: DMSO
 Temperature: 75.0 C
 Spin: 15 Hz
 Result: 7657
 Data: 14926
 Magnitude: 8.45207
 Location: 116913
 Name: 2
 T1: 6.0 s
 T2: 50.0 s
 T3: 100.0 s
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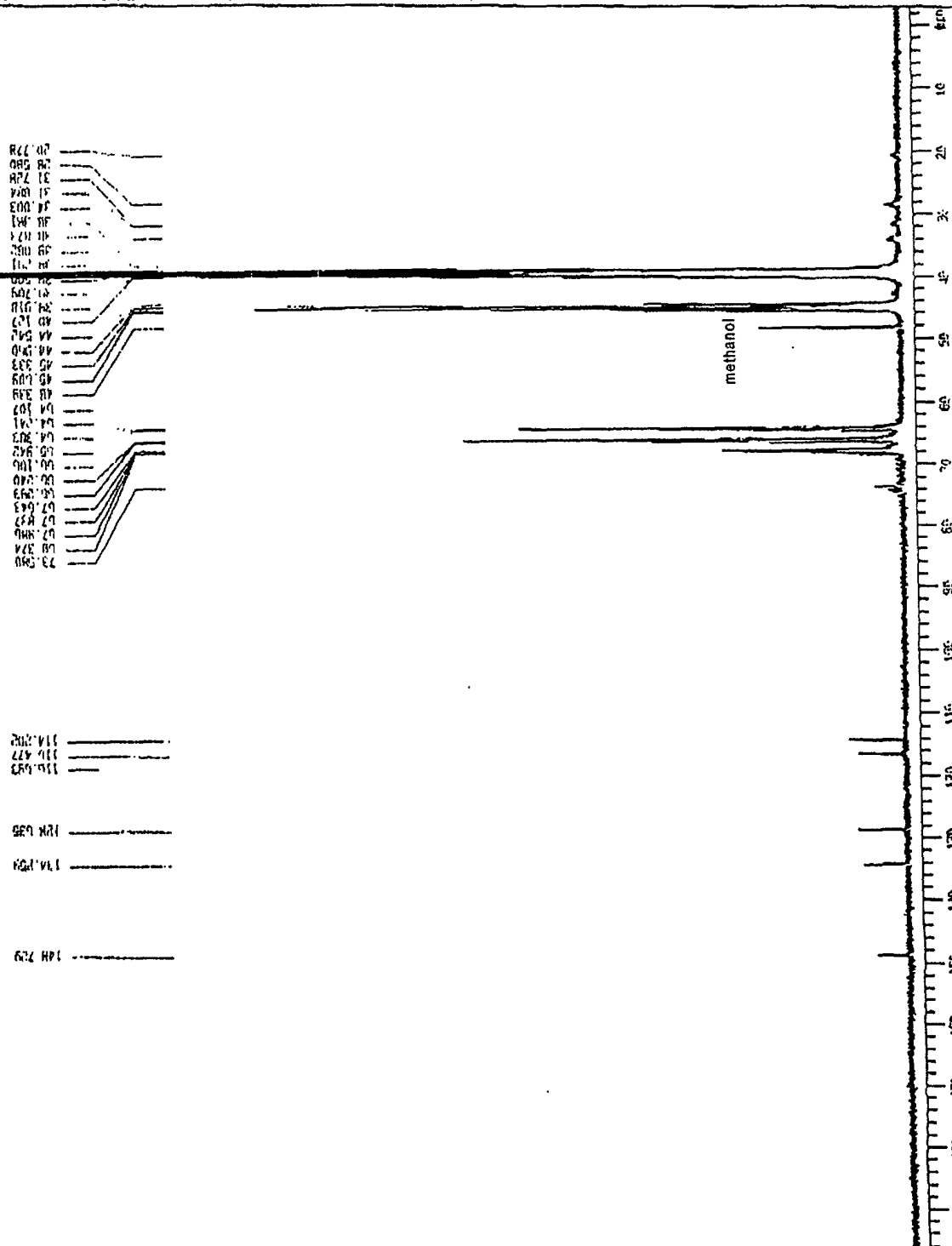
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Chart No. 3-1

